

APPENDIX I
CLAIMS AFTER ENTRY OF FIRST PRELIMINARY AMENDMENT

A) New Independent Claim 62 and Claims Dependent Therefrom

62. In a continuous process for preparing highly esterified polyol fatty acid polyester by interesterifying polyol containing more than four esterifiable hydroxy groups and fatty acid ester of an easily removable alcohol in a heterogeneous reaction mixture wherein said easily removable alcohol is removed from said reaction mixture as the reaction proceeds, the improvement which comprises

- (A) carrying out an initial stage of the interesterifying reaction in a continuous manner under conditions of backmixing suitable for maintaining within said reaction mixture a level of lower partial fatty acid esters of said polyol that is sufficient to emulsify said reaction mixture; and
- (B) carrying out at least a final stage of the interesterifying reaction in a continuous manner under conditions approaching plug-flow conditions after the degree of esterification of said polyol has reached at least about 50%.

14. The process of Claim 62 wherein the initial stage of said interesterifying reaction is carried out under conditions of backmixing until the average degree of esterification of the polyol is from about 20% to about 70%, to thereby provide sufficient lower partial polyol polyester to aid in solubilization of the polyol.

15. The process of Claim 14 wherein the initial stage of said interesterifying reaction is carried out under conditions of backmixing until the average degree of esterification of the polyol is from about 35% to about 60%.

27. The process of Claim 62 wherein, in the initial stage of the interesterifying reaction, the reaction mixture contains soap emulsifier at a level of from about 0.001 to about 0.6 mole per mole of polyol.
28. The process of Claim 27 wherein said soap emulsifier is at a level of from about 0.2 to about 0.4 mole per mole of polyol and said conditions of backmixing are continued until the degree of esterification of said polyol is from about 30% to about 60%.
29. The process of Claim 62 wherein the temperature in the initial stage of the interesterifying reaction is from about 130°C to about 140°C and in the final stages is from about 80°C to about 120°C, wherein the alcohol removed from said reaction mixture is volatile, the pressure in the final stages of the reaction is maintained at from about 15 to about 300 mm Hg and the removal of the volatile alcohol is assisted by increasing the mass transfer area of the reaction mixture.
30. The process of Claim 29 wherein the temperature in the initial stage is from about 132°C to about 135°C and the temperature in the subsequent stages is from about 100°C to about 120°C.
31. The process of Claim 30 wherein the pressure is maintained between about 15 and about 100 mm Hg.
32. The process of Claim 31 wherein said mass transfer area is increased by increasing the surface area of the reactor and/or sparging with an inert gas.
40. The process of Claim 62 wherein the initial catalyst level is from about 0.01 to about 0.1 mole of catalyst per mole of polyol; the initial level of soap emulsifier in the first stage of the interesterifying reaction is from about 0.0001 to about 0.6 mole per mole of polyol; and

the polyol is a solid that has had its particle size reduced by mechanical size reduction and has a particle size of less than about 50 microns.

41. The process of Claim 40 wherein said catalyst level is from about 0.02 to about 0.05 mole per mole of polyol; said level of soap emulsifier is from about 0.2 to about 0.4 mole per mole of polyol; and said polyol is sucrose having a particle size of less than about 10 microns.

42. The process of Claim 40 wherein the molar ratio of said total ester reactant to each said esterifiable hydroxy group of said polyol is from about 0.9:1 to about 1.2:1.

43. The process of Claim 42 wherein said molar ratio of said total ester reactant to each said esterifiable hydroxy group is from about 1:1 to about 1.2:1.

44. The process of Claim 62 wherein the final degree of esterification of said polyol reaches at least about 70%.

45. The process of Claim 44 wherein the final average degree of esterification of the polyol is at least about 95%.

48. The process of Claim 27 wherein the final stages of the reaction are carried out under plug-flow conditions, after the degree of esterification of said polyol has reached at least about 50%.

49. The process of Claim 48 wherein any soap that is insoluble in the reaction mixture is removed by filtration after the degree of esterification reaches at least about 60%.

50. The process of Claim 49 wherein any unreacted polyol, and catalyst having particle sizes above about one micron are removed before the degree of esterification of the polyol reaches about 75%.

51. The process of Claim 50 wherein the molar ratio of said total ester reactant to each said esterifiable hydroxy group of said polyol is from about 1:1 to about 1.2:1.

54. The process of Claim 14 which is carried out in a series of at least two reaction vessels.

55. The process of Claim 54 wherein there are from three to about eight of said reaction vessels.

B) Amended Independent Claim 1 and Claims Dependent Therefrom

1. A continuous process for preparing highly esterified polyol fatty acid polyester by interesterifying polyol containing more than about four esterifiable hydroxy groups and fatty acid ester of easily removable alcohol in a heterogeneous reaction mixture wherein

- a) a catalyst is used in the reaction mixture at an initial level of from about 0.01 to about 0.5 mole of catalyst per mole of polyol;
- b) a soap emulsifier is used in the initial stage of the process at a level of from about 0.001 to about 0.6 mole of soap per mole of polyol;
- c) the molar ratio of total ester reactant to each esterifiable hydroxy group of the polyol in the reaction mixture ranges from about 0.9:1 to about 1.2:1;
- d) the temperature in the initial stage of the process ranges from about 130°C to about 140°C and in the final stages of the process ranges from about 80°C to about 120°C; and
- e) easily removable alcohol is removed from the reaction mixture as the interesterifying reaction proceeds; and

wherein said process utilizes one or more of the following features:

- (1) the polyol is a particulate solid that has had its particle size reduced by mechanical size reduction to a particle size of less than about 100 microns;
- (2) after the degree of esterification is greater than about 60%, and the soap is insoluble in the reaction mixture, the soap is removed from the reaction mixture by filtration or centrifugation in a continuous process;
- (3) unreacted polyol having particle sizes above about one micron is removed, before any soap that is present becomes insoluble, in a continuous process;
- (4) said easily removable alcohol is a volatile alcohol, the pressure above the reaction mixture in the final stages of the interesterifying reaction is maintained at from about 15 to about 300 mm Hg and the removal of the volatile alcohol is assisted by increasing the mass transfer area of the reaction mixture;
- (5) the initial stage of the interesterifying reaction is carried out in a continuous manner under conditions of backmixing suitable for maintaining within said reaction mixture a level of lower partial fatty acid esters of said polyol that is sufficient to emulsify said reaction mixture;
- (6) at least the final stage of the interesterifying reaction is carried out in a continuous manner under conditions approaching plug-flow conditions after the degree of esterification of said polyol has reached at least about 50%.

2. The process of Claim 1 which utilizes Feature (1) wherein the polyol is a particulate solid that has had its particle size reduced by mechanical size reduction and has a particle size of less than about 100 microns.

3. The process of Claim 2 wherein said particle size is less than about 50 microns.
4. The process of Claim 3 wherein said particle size is less than about 10 microns.
5. The process of Claim 1 in which the initial catalyst level is from about 0.01 to about 0.1 mole of catalyst per mole of polyol.
6. The process of Claim 5 in which the catalyst level is from about 0.02 to about 0.05 mole of catalyst per mole of polyol.
8. The process of Claim 1 wherein said initial level of soap emulsifier is from about 0.2 to about 0.4 mole per mole of polyol.
9. The process of Claim 8 wherein said soap emulsifier is a potassium soap of hydrogenated fatty acid containing from about 10 to about 22 carbon atoms.
10. The process of Claim 1 in which: the initial catalyst level is from about 0.01 to about 0.1 mole of catalyst per mole of polyol; the initial level of soap emulsifier in the first stage of the reaction is from about 0.2 to about 0.4 mole of soap per mole of polyol; and the polyol is a solid that has had its particle size reduced by mechanical size reduction to a particle size of less than about 50 microns.
11. The process of Claim 10 wherein said catalyst level is from about 0.02 to about 0.05 mole per mole of polyol; and said polyol is sucrose having a particle size of less than about 10 microns.
13. The process of Claim 10 wherein said molar ratio of said total ester reactant to said esterifiable hydroxy group is from about 1:1 to about 1.2:1.

17. The process of Claim 10 wherein any soap that is insoluble in the reaction mixture is removed by filtration after the degree of esterification reaches at least about 60%.
18. The process of Claim 10 wherein any unreacted polyol and catalyst having particle sizes above about one micron are removed before the degree of esterification of the polyol reaches about 75%.
22. The process of Claim 1 which utilizes Feature (4) wherein the alcohol removed from the reaction mixture is volatile, the pressure in the final stages of the reaction is maintained at from about 15 to about 300 mm Hg and the removal of the volatile alcohol is assisted by increasing the mass transfer area of the reaction mixture.
23. The process of Claim 22 wherein the temperature in said initial stage is between about 132°C. and about 135°C.
24. The process of Claim 23 wherein said pressure is from about 15 to about 100 mm Hg.
56. The process of Claim 1 wherein: the polyol is sucrose having a particle size of less than about 100 microns; the unreacted sucrose is removed after the degree of esterification is at least about 15% and before the soap becomes insoluble; the said initial stage is carried out under conditions of backmixing to maintain a level of lower partial fatty acid esters of said polyol in an emulsifying amount; and the final stages are carried out in a continuous manner under plug flow conditions after the degree of esterification is at least about 50%.
57. The process of Claim 56 wherein the soap emulsifier is removed after the degree of esterification is at least about 60%.

58. The process of Claim 1 wherein any unreacted polyol and catalyst having particle sizes above about one micron are removed while the soap emulsifier is soluble in the reaction mixture and before the degree of esterification of the polyol reaches about 75%.

[A:4233-03]